(FILE 'HOME' ENTERED AT 11:02:26 ON 15 SEP 2003) FILE 'REGISTRY' ENTERED AT 11:02:41 ON 15 SEP 2003 L11 S TEFLON/CN FILE 'CA' ENTERED AT 11:04:06 ON 15 SEP 2003 53895 S EXTRACT? (5A) (VESSEL OR CONTAINER OR CELL OR TUBE) L2105 S L2 AND (L1 OR TEFLON OR PTFE OR POLYTETRAFLUOROETHYLENE OR (POLY OR L3 POLYMER) (1A) TETRAFLUOROEHTYLENE) L41293 S L2 AND FLOW? L5 25 S L3 AND L4 14016 S (FILTRAT? OR FILTER?) (5A) (VESSEL OR CONTAINER OR CELL OR TUBE) L6 179 S L6 AND (L1 OR TEFLON OR PTFE OR POLYTETRAFLUOROETHYLENE OR (POLY OR L7 POLYMER) (1A) TETRAFLUOROEHTYLENE) L8 32 S L7 AND PRESSUR? L9 57 S L5, L8 L1053 S (L9 NOT PY>2000)OR(L9 AND PATENT/DT) => d l10 bib, ab 1-53 L10 ANSWER 9 OF 53 CA COPYRIGHT 2003 ACS on STN ΑN 134:133646 CA Lined sampling vessel including a filter to separate solids from liquids on TIIN Shurtliff, Rodney M.; Klingler, Kerry M.; Turner, Terry D. Bechtel Bwxt Idaho, Llc, USA PA SO U.S., 12 pp. PΙ US 6187209 B1 20010213 US 2000-533462 20000323 PRAI US 2000-533462 20000323 A filtering app. has an open canister with an inlet port. A canister lid is provided which includes an outlet port for the passage of fluids from the canister. Liners are also provided which are shaped to fit the interiors of the canister and the lid, with at least the canister liner preferably being The sample to be filtered is positioned inside the canister liner, with the lid and lid liner being put in place thereafter. A filter element is located between the sample and the outlet port. Seals are formed between the canister liner and lid liner, and around the outlet port to prevent fluid leakage. A pressure differential is created between the canister and the canister liner so that the fluid in the sample is ejected from the outlet port and the canister liner collapses around the retained solids. L10 ANSWER 13 OF 53 CA COPYRIGHT 2003 ACS on STN AN132:121580 CA Flow injection Fourier transform infrared determination of caffeine in TIΑU Bouhsain, Z.; Garrigues, J. M.; Garrigues, S.; de la Guardia, M. Department of Analytical Chemistry, University of Valencia, Valencia, Spain CS Vibrational Spectroscopy (1999), 21(1-2), 143-150 A fully automated procedure was developed for the FTIR spectroscopic detn. SO AΒ of caffeine in coffee samples. The method involves the online extn. of caffeine with CHCl3. Samples, weighed inside empty PTFE cartridges of 0.5 cm internal diam. (internal diam.) and 1.5 mL vol., were humidified with four drops of 0.25M NH3. The cartridge was installed in a flow manifold, in which samples were extd. in a closed-flow system with 1 mL CHCl3 during 6 Four hundred microliters of the ext. were introduced in a microflow cell and absorbance measured as a function of time at 1659 cm-1, with a baseline established between 1900 and 830 cm-1, thus providing a diagram.

Peak height values of the FI recordings, obtained for samples, were interpolated in an external calibration line established from std. solns. of caffeine in CHCl3. The method provided a limit of detection (LOD) of 9 mg L-1 caffeine, a relative std. deviation of 0.6% for five independent measurements of a soln. contg. 1 mg ml-1 and a sampling frequency of the whole procedure of 6 h-1. Results obtained for market samples agree well with those found by the official chromatog.-spectrometric method, but involving a drastic redn. of solvents, from the 200 mL ether and 50 mL CHCl3 required for each sample prepn. by the ref. procedure to <30 mL CHCl3 necessaries for the whole detn. of caffeine in a sample, also including stds. and carrier soln.

- L10 ANSWER 16 OF 53 CA COPYRIGHT 2003 ACS on STN
- AN 131:96498 CA
- TI Method performance of the closed vessel microwave-assisted acid extraction using 50% HNO3:HCl (3:2) with positive pressure Teflon membrane filtration
- AU Robbat, Albert, Jr.; Simpson, Robert L., III
- CS Department Chemistry, Tufts University, Medford, MA, 02155, USA
- SO Fresenius' Journal of Analytical Chemistry (1999), 364(4), 305-312
- AB The recovery from soil of 22 metals on the U.S. Environmental Protection Agency's (EPA) Target inorg. analyte list is described. The extn. method was developed to provide a safe, rapid, and anal. reliable means of leaching metals from soils and sediments in one procedure. The influence of digestion matrixes, filtration media, ref. std. types, and instrument performance of ICP-AES is presented. The method employs a closed vessel, temp. and pressure controlled, microwave acid digestion using 20 mL of 50% HNO3: HCl in a ratio of (3:2). The digestate was filtered through a pos. pressure Teflon membrane cartridge. This procedure recovered all metals at concns. equal to or greater than what is possible by EPA standardized methods or other methods published in the literature. Excellent method precision and accuracy was obtained for all metals, esp. Ag and Sb. The pos. Teflon membrane filtration system yielded higher and statistically different concns. of Mn, Zn, Cu, Fe, As, Cd, Pb, Ag, and Sb than paper filtration in half the time. These findings were produced from std. ref. soils and soil collected from a hazardous waste site landfill.
- L10 ANSWER 23 OF 53 CA COPYRIGHT 2003 ACS on STN
- AN 120:326304 CA
- TI Automated supercritical fluid extraction system
- IN Houck, Raymond K.; Koebler, Douglas J.; Williams, Glen P.; Kato, Kenneth J.;
  Parks, Robert D.; Bauer, Paul A., Jr.
- PA Suprex Corp., USA
- SO PCT Int. Appl., 110 pp.
- PI WO 9408683 A1 19940428 WO 1993-US9680 19931012 US 5866004 A 19990202 US 1995-524916 19950908
- PRAI US 1992-962463 A 19921016
- AB A supercrit. fluid extn. system for extg. analytes from samples comprises a holding means, and a moving means which selectively moves extn. vessels into an extn. chamber; the system also includes a computer for controlling the extn., and a collecting means having a restrictor for controlling the flow of analyte. The system can be used to analyze polyarom. hydrocarbons in soil.
- L10 ANSWER 25 OF 53 CA COPYRIGHT 2003 ACS on STN
- AN 120:137791 CA
- TI Apparatus for supercritical fluid extraction
- IN Ichinomya, Makoto; Kato, Osamu; Nishida, Hideo; Yutaka, Hideki; Takenaka,
  Mamoru

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PA
     Kobe Steel Ltd, Japan
     Jpn. Kokai Tokkyo Koho, 10 pp.
SO
     JP 05305202
                             19931119
PΙ
                       A2
                                            JP 1992-111600
                                                              19920430
PRAI JP 1992-111600
                             19920430
     In a supercrit. fluid extn. app. comprising an external vessel and an
     inserted internal vessel for contg. substance to be treated by supercrit.
     fluid (e.g., CO2) flowing in vertical direction, sealing member(s) (e.g.,
     PTFE) is arranged between the outer- and inner circumferential surfaces of
     the internal- and external vessel adjacent to their upper (or lower) end,
     and guide member(s) softer than the 2 vessels is arranged between the outer-
     and inner circumferential surfaces of the internal- and external vessel
     adjacent to their lower (or upper) end for preventing contacting of the 2
     vessels. The sealing member(s) has tensile strength ≥250 kg/cm2, and gas
     permeability \geq 10000 \text{ x } 10-10 \text{ cm} 3-\text{mm}/(\text{cm} 2-\text{s-cmHg}). The app. is used for extn.
     of desired substances from solid substances, e.g., natural products, etc.
L10
     ANSWER 42 OF 53 CA COPYRIGHT 2003 ACS on STN
ΑN
     102:181779
     Determination of sulfur in biological samples by vacuum-ultraviolet
TI
     inductively-coupled plasma atomic emission spectrometry
ΑU
     Morita, Masatoshi; Uehiro, Takashi; Fuwa, Keiichiro
CS
     Natl. Inst. Environ. Stud., Tsukuba, 305, Japan
     Analytica Chimica Acta (1984), 166, 283-8
SO
     Std. ref. materials (e.g., serum, hair, liver, mussel, Chlorella, and
AB
     orchard leaves) were decompd. by high-pressure bomb digestion in a Teflon
     container with HNO3, dild. with water, filtered, and the absorbance was
     measured at 180.7 nm by the title method for S detn.. The detection limit
     was \sim 10 \mu \text{g S/L}.
                      The results were satisfactory.
=> loq y
STN INTERNATIONAL LOGOFF AT 11:14:49 ON 15 SEP 2003=> d his
     (FILE 'HOME' ENTERED AT 16:41:12 ON 11 SEP 2003)
     FILE 'CA' ENTERED AT 16:41:57 ON 11 SEP 2003
          E MANGANINI S/AU
       17 S E3-7
L1
          E DOHERTY K/AU
L2
        6 S E8, E17
L3
       22 S L1-2
L4
        1 S L1 AND L2
L5
     6496 S MICROWAVE (6A) (PROCESSING OR REACTION OR VESSEL OR CONTAINER OR
          DIGEST?)
L6
     1369 S L5 AND (SAMPLE OR SOIL OR SOLID) (6A) (PREPAR? OR DIGEST? OR REACTI? OR
          FRACTIONA? OR EXTRACT?)
L7
       45 S L6 AND (FILTER OR MEMBRANE OR POROUS)
L8
      146 S L6 AND (TEFLON OR PTFE OR PFA OR ULTEM OR POLYTETRAFLUOROETHYLENE)
L9
      182 S L4, L7-8
L10
      146 S L9 NOT PY>2000
L11
        2 S L9 NOT L10 AND PATENT/DT
L12
      148 S L10-11
=> d bib, ab 1-148 l12
L12
     ANSWER 1 OF 148
                     CA COPYRIGHT 2003 ACS on STN
AN
     138:162651 CA
TΙ
     Sequential processing reaction vessel for chemical fractionation and
```

Manganini, Steven J.; Doherty, Kenneth W.; Hammer, Terence R.; Lancaster,

IN

Bruce A.

PA USA

SO U.S. Pat. Appl. Publ., 13 pp.

PI US 2003031600 A1 20030213 US 2001-771354 20010125

PRAI US 2000-177837P P 20000125

- AΒ A sequential processing reactor vessel and method is disclosed for accelerated extn. and fractionation of chem. analytes from complex solid sample materials. The device and method provide for sequential extn. of elemental constituents from solid materials by sequentially contacting target samples within a single reaction vessel using different reagents at temps. ≤150°. and pressures up to 150 psi to accelerate reactions. aggressive chem. treatments provided by the disclosed device and method provide for complete digestion of solid samples in liq. analyte samples that can be directly analyzed by conventional spectrometry or other suitable methods. The disclosed device and method provide for efficient sample processing and accelerated reactions to significantly reduce processing times and costs for elemental anal. of solids while improving accuracy, precision and reliability of results compared to conventional devices and The disclosed device and method are compatible with both conventional convection and radiant heating sources as well as microwave heating and can be readily adapted to marine, geol., environmental, industrial and research solids anal. applications.
- L12 ANSWER 14 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 132:269115 CA

- TI Sample preparation and determination of the element content from low-weight feather samples
- AU Vallner, Judit; Posta, J.; Szep, T.; Braun, M.; Balogh, A.; Kiss, F.
- CS Department of Environmental Science, Gyorgy Bessenyei College, Nyiregyhaza, 4400, Hung.
- SO Toxicological and Environmental Chemistry (1999), 70(3-4), 297-304
- AB In examg, heavy metals in bird feathers, birds must be protected by using as little material as possible. However, small sample size has generated difficulties in sample prepn. Ultrasonic washing was shown to be efficient in eliminating all water-sol, contamination from the surface of feather samples. Ways to avoid previous contamination from the walls of a Teflon bomb during microwave digestion are discussed. There is no loss of sample, and the memory effect of the bombs is eliminated. Elemental content (Ca, Fe, K, Mg, Mn, Na, Sr) of a homogenized feather powder sample of the wt. of 1 swallow feather was detd. using inductively coupled plasma at. emission spectrometry with impulse sample introduction.
- L12 ANSWER 15 OF 148 CA COPYRIGHT 2003 ACS on STN

132:267822 CA

ΑN

- TI Rapid determination of Os isotopic composition by sparging OsO4 into a magnetic-sector ICP-MS
- AU Hassler, D. R.; Peucker-Ehrenbrink, B.; Ravizza, G. E.
- CS MIT/Woods Hole Oceanographic Institution (WHOI) Joint Program in Oceanography, Woods Hole, MA, USA
- SO Chemical Geology (2000), 166(1-2), 1-14
- AB A method is presented for the rapid detn. of Os isotopic compns. and platinum group element (PGE) concns. using ICP-MS, in the same sediment sample split. The method makes use of transfer of volatile OsO4 by an Ar gas stream into the torch of a magnetic sector ICP-MS for anal. A variety of partial or complete sample dissoln. methods can be used, namely (1) microwave oven digestion of samples in pressurized Teflon vessels under oxidizing acidic conditions, (2) NiS fire assay for complete sample dissoln. and PGE preconcn., (3) acid leaching techniques to ext. labile PGE

fractions, and (4) Carius tube digestion. Microwave oven sample digestion with HF-HNO3-HCl in an all-Teflon system as well as Carius tube digestion also allows the detn. of Os isotopic compn. and Os and Re concns. on the same sample split. Spike-sample equilibration can be monitored online during sparging into the ICP-MS. The elimination of a nebulizer for Os isotope ratio measurements minimizes memory problems that are often assocd. with the detn. of Os isotope ratios in liq. samples by ICP-MS. The external reproducibility of 1870s/1880s measurements of an inhouse Os std. soln. using a single-collector, magnetic sector ICP-MS (Finnigan ELEMENT) is 0.78% (1 S.D., n=13) for analyte amts. ranging from 81 pg to 1.22 ng total Os. The ion yield for 7-min data acquisitions is ~5 x 10-5. Compared to neg. thermal ionization mass spectrometry (N-TIMS), sample throughput is about on an order of magnitude higher. If NiS fire assay is used for PGE preconcn., complementary PGE can be detd. in the liq. residue by ICP-MS using a microconcentric nebulizer after the extn. of OsO4.

- L12 ANSWER 17 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 132:147685 CA
- TI Usefulness of enzymatic hydrolysis procedures based on the use of pronase E as sample pre-treatment for multi-element determination in biological materials
- AU Bermejo-Barrera, Pilar; Fernandez-Nocelo, Susana; Moreda-Pineiro, Antonio; Bermejo-Barrera, Adela
- CS Faculty of Chemistry, Nutrition and Bromatology, Department of Analytical Chemistry, University of Santiago de Compostela, Santiago de Compostela, 15706, Spain
- SO Journal of Analytical Atomic Spectrometry (1999), 14(12), 1893-1900
- AB Several minor (Cu, Fe, Mg and Zn) and trace (Ag, As, Cd and Pb) elements were extd. from biol. materials, such as human hair and mussel, using an enzymic hydrolysis procedure based on pronase E. The reaction conditions, viz., pH and temp., were fixed at optimum values of 7.4 and 37°, resp. Other variables affecting the enzymic hydrolysis procedure, such as enzymic hydrolysis time, enzyme concn., vol. of enzyme soln. and sample mass, were studied and optimized. The pH value was adjusted with a TRIS-HCl soln. as buffer system. The minor elements were measured by FAAS while trace elements were detd. by ETAAS under optimum conditions. In order to det. the total element concn. in samples, a microwave-induced acid digestion procedure in lab.-made low pressure PTFE bombs was optimized. The enzymic hydrolysis was effective for mussel samples (recoveries of about 100% were obtained for As, Cd, Cu and Mg); however, it was poor for human hair (recoveries were lower than 70%).
- L12 ANSWER 19 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 131:348663 CA
- TI Sample preparation by focused microwave digestion for ICP-MS
- AU Curdova, Eva; Koplik, Richard
- CS VSCHT, Prague, 16628, Czech Rep.
- SO CHEMagazin (1999), 9(5), 15-16
- LA Czech
- Microwave-assisted pressure digestion is a well established method of sample prepn. for trace element anal. A new generation of focused microwave decompn. units is represented by the Polish UniClever system. Sample decompn. takes place in a teflon (TFM-PTFE) vessel under controlled pressure. Temp. and pressure valves are monitored in each individual unit. The whole system can be controlled by a personal computer. Microwave digestion in the UniClever system using HNO2+H2O2 mixt. was used for the decompn. of several certified ref. materials of plant origin (alfalfa, rye grass, pine needles, apple leaves). The final detn. of trace elements (Cd,

Cu, Pb, Zn) was accomplished by ICP-MS. The results proved a good efficiency of the sample decompn. and excellent accuracy.

- L12 ANSWER 20 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 131:256449 CA
- TI Microwave-Enhanced Flow System for High-Temperature Digestion of Resistant Organic Materials
- AU Pichler, Ulrike; Haase, Anja; Knapp, Guenter; Michaelis, Markus
- CS Institute of Analytical Chemistry Micro- and Radiochemistry, Technical University Graz, Graz, A-8010, Austria
- SO Analytical Chemistry (1999), 71(18), 4050-4055
- Microwave-assisted flow digestion systems open up new possibilities in fully AΒ automated sample prepn. for element anal. For an extensive and fast oxidn. of org. materials with nitric acid, temps. of more than 200°C are necessary. To achieve the desired temps. of ~250°C, it is essential that the pressure in the system can be increased up to 35 bar. The teflon tubes used, however, do not withstand the vapor pressure of the digestion mixt. at these temp. levels. A high-pressure flow digestion device is described that enables the application of such high temps. by means of a novel pressure equilibration system. PTFE or PFA tubes can be used up to 250°C if no mech. stress is applied to the tube wall. The pressure equilibration system keeps the pressure inside and outside the digestion tube equal even for extremely fast oxidn. reactions. The digestion of easy, medium, and difficult oxidizable substances (glucose, glycine, and phenylalanine, resp.) shows the importance of digestion temps. around 250°C. The extremely violent digestion of glucose with concd. nitric acid can be carried out as easy as the difficult oxidn. of phenylalanine by means of this system. 2 (defatted lobster hepatopancreas tissue) was digested under different conditions to indicate the high oxidn. capabilities in comparison with a com. medium-pressure flow digestion device. The anal. of the SRMs milk powder (BCR 063, BCR 151), bovine liver (BCR185), and pig kidney (BCR 186) after digestion at 245°C and 5 min showed good agreement with the certified values.
- L12 ANSWER 25 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 131:115534 CA
- TI Presence and distribution of arsenical species in beers
- AU Herce-Pagliai, C.; Gonzalez, G.; Camean, A. M.; Repetto, M.
- CS Departmento de Bioquimica, Bromatologia y Toxicologia, Universidad de Sevilla, Seville, 41012, Spain
- SO Food Additives and Contaminants (1999), 16(6), 267-271
- The total content of arsenic and of its inorg. (As(III) and As(V)) and org. AB (monomethylarsonic acid, MMAA, and dimethylarsinic acid, DMAA) species were detd. in a set of 21 alc. and alc.-free beer samples using the technique of Hydride Generation At. Absorption Spectrometry. For total arsenic anal., beer samples were dried and then microwave digested with nitric acid in polytetrafluoroethylene containers. For the speciation anal., beers were previously subjected to ion exchange chromatog. to elute the mentioned inorg. and org. arsenical species. Both microwave digestion and chromatog. sepn. methods were validated from certified ref. materials and prepd. std. solns., resp. The results obtained are presented in terms of the distribution and occurrence of arsenical species in the samples. levels of the beer samples were in the range of 1.5-12.4  $\mu$ g/L. influence of the prodn. process for the alc.-free beers in the speciation of arsenic is discussed. In alc. beers MMAA was the most abundant species, and for non-alc. beers inorg. As(III) was similar to the org. species. An estd. intake of total As of 0.47  $\mu g/person/day$  and 11.4  $\mu g/person/day$  was obtained for av. consumers and for heavy drinkers, resp.

- L12ANSWER 26 OF 148 CA COPYRIGHT 2003 ACS on STN
- 131:96498 CA AN
- TIMethod performance of the closed vessel microwave-assisted acid extraction using 50% HNO3:HCl (3:2) with positive pressure Teflon membrane filtration
- Robbat, Albert, Jr.; Simpson, Robert L., III ΑU
- CS
- Department Chemistry, Tufts University, Medford, MA, 02155, USA Fresenius' Journal of Analytical Chemistry (1999), 364(4), 305-312 SO
- AΒ The recovery from soil of 22 metals on the U.S. Environmental Protection Agency's (EPA) Target inorg. analyte list is described. The extn. method was developed to provide a safe, rapid, and anal. reliable means of leaching metals from soils and sediments in one procedure. The influence of digestion matrixes, filtration media, ref. std. types, and instrument performance of ICP-AES is presented. The method employs a closed vessel, temp. and pressure controlled, microwave acid digestion using 20 mL of 50% HNO3: HCl in a ratio of (3:2). The digestate was filtered through a pos. pressure Teflon membrane cartridge. This procedure recovered all metals at concns. equal to or greater than what is possible by EPA standardized methods or other methods published in the literature. Excellent method precision and accuracy was obtained for all metals, esp. Aq and Sb. pos. Teflon membrane filtration system yielded higher and statistically different concns. of Mn, Zn, Cu, Fe, As, Cd, Pb, Ag, and Sb than paper filtration in half the time. These findings were produced from std. ref. soils and soil collected from a hazardous waste site landfill.
- ANSWER 27 OF 148 CA COPYRIGHT 2003 ACS on STN L12
- AN131:22488 CA
- Determination of chromium in airborne particulate matter by high resolution TI and laser ablation inductively coupled plasma mass spectrometry
- ΑU Wang, Chu-Fang; Chin, Ching-Jer; Luo, Shen-Kay; Men, Lee-Chung
- Department of Nuclear Science, National Tsing Hua University, Hsinchu, CS Taiwan
- SO Analytica Chimica Acta (1999), 389(1-3), 257-266
- AΒ An optimized sample digestion procedure to det. Cr in airborne particulate matter by inductively coupled plasma mass spectrometry (ICP-MS) is discussed. High resoln. (HR) ICP-MS is used to examine how the added acid mixt. and subsequent evapn. process affect spectroscopic interference. Exptl. results indicated a 2-step high pressure bomb acid digestion (first step with HNO3 + HClO4; second step with HF) procedure without evapn. is the optimum pretreatment procedure to det. Cr by ICP-MS. In addn., laser ablation (LA) ICP-MS is used directly to analyze for Cr in airborne particulate matter. Results obtained by LA-ICP-MS correlated well with certified values and values obtained from the conventional acid digestion/HR-ICP-MS method. The Cr detection limit in air particles by HR-ICP-MS with the proposed digestion method is <10 ng/q. Furthermore, LA-ICP-MS provides a rapid, direct anal. technique with a detection limit of 0.05  $\mu g/filter.$
- CA COPYRIGHT 2003 ACS on STN L12ANSWER 31 OF 148
- AN 130:109334 CA
- Optimized microwave digestion procedure for cadmium analysis of mussel TIsamples
- ΑU Yebra, M. C.; Enriquez, M. F.
- CS Department of Analytical Chemistry, Nutrition and Bromatology, Chemistry Faculty, Univ. of Santiago, Santiago de Compostela, 15706, Spain
- SO Analusis (1998), 26(7), 261-263
- A simple and rapid acid sample digestion method by microwave heating in AΒ high-pressure teflon bombs is reported for the detn. of cadmium in mussels by flame at. absorption spectrometry (FAAS) coupled online with a flow-

injection preconcn. system. Tests concerning the **digestion** time, the power of the **microwaves** and the amt. of nitric acid were studied with the National Research Council of Canada lobster hepatopancreas marine (Tort-1) as certified ref. material. Using 2 min of digestion time and 2 mL of nitric acid, the complete dissoln. of 0.2 g of freeze-dried sample is possible. The anal. results, obtained with calibration graph as well as by the std. addn. method, were agreed well with the certified value of the ref. material. This procedure was applied to the detn. of cadmium in mussel samples from estuaries in Galicia, Spain.

- L12 ANSWER 34 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 130:42756 CA
- TI Chemical characterization of municipal solid waste incineration residue: dissolution of elements with a microwave-dilute acids digestion technique compared to conventional methods
- AU Dugenest, S.; Olle, M.; Ribes, A.; Grenier-Loustalot, M. F.
- CS Service Central d'Analyse, Centre National de la Recherche Scientifique, Vernaison, 69390, Fr.
- SO Analusis (1998), 26(7), 256-260
- The aim of this work was to evaluate the focused microwave technique in an open system for the rapid dissoln. of elements in fly ash and **filter** cakes, in comparison to conventional wet and dry methods. The method involved 15 min of heating in microwave system in 2 N nitric or hydrochloric acid. It was tested on different types of samples and led to the dissoln. of Ca, Na, K, Zn, Pb, As with 2 N HNO3 or HCl with Cd. Hg with 2 N HNO3 for subsequent detn. by at. emission or absorption spectrometry. Thereby this method represents a rapid **sample prepn**. technique for chem. analyses.
- L12 ANSWER 35 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 129:269679 CA
- TI Combined vessel system used with microwave decomposing apparatus
- IN Kubovics, Ferenc
- PA Hung.
- SO PCT Int. Appl., 16 pp.
- PI WO 9845680 A1 19981015 WO 1998-HU30 19980324
- PRAI HU 1997-736 19970404
- AB The invention relates to a combined **vessel** system used with **microwave** decompg. app. comprising a basic **vessel** placed on a rotary plate for receiving the material to be decompd. the sample and chem. agents used for decompn. In the combined vessel system the basic vessel is formed to be suitable for a combined microwave decompn. The basic vessel is provided with a bell-shaped refrigerating cover closing the basic vessel from above and having an aperture on its end opposite to the basic vessel. Said aperture of the bell-shaped refrigerating cover is connected with a vacuum pipe draining the gases developed during the decompn.
- L12 ANSWER 40 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 128:42984 CA
- TI Sample decomposition-Quo vadis
- AU Knapp, Gunter
- CS Department Analytical Chemistry Micro- and Radiochemistry, Technical University Graz, Graz, Austria
- Contemporary State and Trends of Decomposition Methods in Analytical Chemistry, Proceedings, Slovak-Austrian Symposium, Kosice, Slovakia, Feb., 1997 (1997), 10-17. Editor(s): Krakovska, Erika. Publisher: Stroffek Publishing, Kosice, Slovakia.
- AB A review, with 12 refs., is given on **sample prepn**. techniques particularly decompn. **Sample** decompn. is the most significant **sample prepn**. technique

for trace element anal. Important functions of sample digestion are dissoln. of solid materials and oxidn. of org. matrix constituents. detn. of elements in the trace and ultratrace range make great demands on decompn. methods. With respect to correct anal. results the classical decompn. techniques fusion, oxidn. with oxygen and wet digestion were improved to reduce systematic errors caused by contamination or losses of elements. In anal. chem. quality control becomes more significant increasingly. But presently it is applied however, 1st of all to measuring techniques and not to sample prepn. One of the most important sample prepn. methods in element anal. is microwave assisted wet digestion in closed pressurized vessels. The parameters important for quality control are in this case pressure and temp. Pressure control is important because the closed vessels are tight only up to a certain pressure. During the decompn. procedure this pressure may be exceeded under no circumstances. The temp., on the contrary, is responsible for the efficiency of the decompn. reaction. The higher the temp., the higher is the oxidn. potential of the digestion reagent. Recording the time temp. curve of the decompn. process the extent of sample degrdn. can be established afterwards. New developments enable recording of pressure and temp. of all vessels in a microwave oven during digestion. Microwave assisted flow digestion systems open up new possibilities in fully automated sample prepn. for element anal. For fast and almost complete sample digestion the temp. must be as high as possible. For this reason elevated pressure has to be applied within the flow digestion systems. For an extensive oxidn. of org. sample constituents with nitric acid temps. of >220° are necessary. Teflon tubes used, however, do not withstand the vapor pressure of the digestion mixt. at 220° or more. Thus new alternatives has to be found to overcome this limitation.

- L12 ANSWER 42 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 128:11532 CA
- TI Development of high-pressure closed-vessel systems for microwave-assisted sample digestion
- AU Matusiewicz, Henryk
- CS Department of Analytical Chemistry, Politechnika Poznanska, Poznan, 60-965, Pol.
- SO Microwave-Enhanced Chemistry (1997), 353-369. Editor(s): Kingston, H. M.; Haswell, Stephen J. Publisher: American Chemical Society, Washington, D. C.
- AB A review with 61 refs. The historical development of high-pressure closedvessel systems in microwave-assisted sample digestion is presented. The
  current state of the art, including advantages and limitations of this
  approach, is discussed. The construction and methodol. controls for a
  series of special microwave-digestion bombs and a universal complex
  microwave-digestion system for chem. prepn. for transferring the sample into
  a liq. state are described. Emphasis is laid on the completeness of the
  digestion, for which the use of high-pressure high-temp. focused-microwaveheated Teflon bomb is prerequisite. The novel prototype system uses focused
  microwaves, operated at 2.45 GHz, to improve digestion capability.
  Methodol. was developed using powd. biol. ref. material. With this new
  decompn. device, org. material is totally oxidized with nitric acid in a
  single-step procedure.
- L12 ANSWER 45 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 127:85671 CA
- TI Optimized microwave preparation procedure for the elemental analysis of aquatic sediment
- AU Wen, Xiang Hua; Wu, Ling Zheng; Zhang, Yun; Chu, Yang
- CS Reasearch Center Eco-Environmental Sciences, Chinese Academy Sciences, Beijing, 100085, Peop. Rep. China

- SO Fresenius' Journal of Analytical Chemistry (1997), 357(8), 1111-1115 AΒ This paper summarizes several key points in applying the microwave prepn. technique to the elemental anal. of aquatic sediments and reports systematic expts. in searching for an optimum microwave prepn. procedure for element anal. in sediment samples. The detn. of the elements Cu, Pb, and Cd in a std. ref. aquatic sediment sample (CRM 280, COMEUR) was achieved by 1st digesting the samples in a microwave oven equipped with PFA advanced composite vessels, followed by AAS measurement. The influence of microwave power, digestion time, various dissoln. reagents, and the HF removing conditions were studied. For a 0.1 g sediment sample the optimum microwave prepn. conditions were: 4-5 mL HNO3/HF/H2O2 as solvent, digesting time 30 min with 100% microwave power, and evapg. the residual acid within 8 min in an open vessel at 80°. The element recovery rates with AAS measurement were ≤92.4-100.6%.
- L12 ANSWER 47 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 127:44048 CA
- TI Determination of mercury in biological and environmental samples by inductively coupled plasma mass spectrometry with the isotope dilution technique
- AU Yoshinaga, Jun; Morita, Masatoshi
- CS National Institute Environmental Studies, Tsukuba, 305, Japan
- SO Journal of Analytical Atomic Spectrometry (1997), 12(4), 417-420
- The accurate and precise detn. of total mercury (Hg) in biol. and AB environmental samples by isotope diln.-inductively coupled plasma mass spectrometry (ID-ICP-MS) is described. The precision of Hg isotope ratio (e.g., 200Hg/202Hg) measurements at the 20 ppb level was <0.5%. deviation of the measured isotope ratios in a std. Hq soln. was <0.5% from the natural ratios. Neither spectral interferences nor matrix effects affect the accuracy and precision of the proposed Hg isotope ratio anal. Total Hg concns. in human hair and sediment CRMs were detd. by ID-ICP-MS after 202Hg addn. and acid decompn. of the samples. Solvent extn. and backextn. was used for sediment anal. The various decompn. methods used for hair samples, i.e., microwave digestion and the Teflon vessel double digestion method, did not give any difference in the anal. value. ICP-MS results were better than those obtained with std. addns. or internal standardization in terms of accuracy and precision. Anal. results for human hair and sediment CRMs were in good agreement with the certified/ref. values.
- L12 ANSWER 48 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 127:8843 CA
- TI Membrane solid-phase extraction (SPE) with closed-vessel microwave elution for the determination of phenolic compounds in aqueous matrixes
- AU Chee, Kok Kay; Wong, Ming Keong; Lee, Hian Kee
- CS Department Chemistry, National University Singapore, Singapore, 119260, Singapore
- SO Mikrochimica Acta (1997), 126(1-2), 97-104
- AB Preconcn. of phenolic compds. was carried out with Empore C18 membrane disks, which were eluted with a closed-vessel microwave extn. (CVME) system. The optimum microwave-elution conditions were obtained by optimizing the elution solvent, elution temp., duration of elution, and vol. of elution solvent. The recoveries of 11 phenolic compds. spiked at 10 and 500 μg/L levels into water, using the optimum conditions, were all >85% with RSD 4.0-10.0%, except for phenol and 4-nitrophenol. Extn. of an industrial wastewater sample suspected of contg. phenolic compds. by SPE-CVME showed similar recoveries of phenol (the only phenolic compd. detected) when compared with LLE and C18 SPE cartridge techniques. Anal. of phenolic

compds. was carried out using liq. chromatog. coupled to a UV detection system.

- L12 ANSWER 49 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 126:350942 CA
- TI Evaluation of rapid **solid sample extraction** using the **microwave**-assisted process (MAP) under closed-**vessel** conditions
- AU Li, Ken; Belanger, Jacqueline M. R.; Llompart, Maria P.; Turpin, Rodney D.; Singhvi, Raj; Pare, J. R. Jocelyn
- CS Microwave-Assisted Processes Division, Environment Canada, Environmental Technology Centre, Ottawa, ON, K1A OH3, Can.
- SO Spectroscopy (Amsterdam) (1997), Volume Date 1996-1997, 13(1), 1-13
- The applicability of MAP extn. and subsequent GC/MS detn. of mixts. of representative toxic substances including PAH, PCB, base-neutrals, chlorinated pesticides and substituted phenols was evaluated. Typical environmental matrixes including sand, soil and air filters were spiked, extd. and analyzed. Recoveries from these matrixes were acceptable (>80 %); precision was generally in the 10% (relative std. deviation) range. The method was then validated using std. ref. materials of marine sediments, harbor sediments and a creosote contaminated soil certified with PAH and PCB. Split samples were also obtained from a contract lab. and the consensus results were used to evaluate recoveries of PAH and organochlorine pesticides. The potential problem of degrdn. of thermally labile pesticides was addressed by measuring degrdn. products of DDT and Endrin after MAP extn. Spiked samples before and after the high temp./high pressure extn. process did not result in addnl. decompn. products.
- L12 ANSWER 50 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 126:342911 CA
- TI Total elemental analysis digestion method evaluation on soils and clays
- AU Wilson, M. A.; Burt, R.; Lynn, W. C.; Klameth, L. C.
- CS Soil Survey Lab., USDA Natural Resources Conservation Service, Lincoln, NE, 68508-3866, USA
- Communications in Soil Science and Plant Analysis (1997), 28(6-8), 407-426 SO ABThe std. digestion method for total elemental anal. of soil material by the Soil Survey Lab. (USDA Natural Resources Conservation Service) uses a 2-mL clay suspension with hydrofluoric acid (HF) in a closed Teflon digestion vessel (method HF-SUS). The accuracy and efficiency of elemental recovery by method HF-SUS was compared to: (a) modification of HF-SUS by use of a dried sample (method HF-DRI); (b) modification of HF-SUS by use of dried sample and HF + aqua regia (method HF + AR); (c) sample digestion by Li metaborate fusion (method FUS); and (d) microwave digestion of samples with HF + aqua regia in Teflon bombs (method MICRO). Three replications of three std. ref. materials (SRMs), fine-earth (<2 mm) from 12 soils, and the clay (<2  $\mu$ m) from 10 of those soils were analyzed. Method HF + AR shows the most consistent statistical agreement with the certified SRM values. Anal. of variance (ANOVA) indicates significant effects ( $\alpha$ =0.05) for method of digestion, nonsignificant effects for method times SRM and method times clay, but significant effects for method times fine-earth. Compn. and/or variability of material are significant factors in the method of digestion. Method HF + AR yields significantly higher exptl. means of Al203, Fe203, and K2O contents and oxide recovery (summation of exptl. means for oxides of all reported elements) than all other methods.
- L12 ANSWER 51 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 126:287237 CA
- TI A generalized method for the determination of nickel in different samples by ETAAS after rapid microwave-assisted digestion

- AU Chakraborty, Ruma; Das, Arabinda K.; Cervera, M. Luisa; De La Guardia, Miguel
- CS Department of Chemistry, University of Burdwan, Burdwan, 713 104, India
- SO Analytical Letters (1997), 30(2), 283-303
- AB Detn. of nickel was carried out by electrothermal atomization at. absorption spectrometry (ETAAS) using unpyrocoated tubes. A generalized method was developed for quant. detn. of total nickel in different types of samples: geol., like rock, soil and sediment, environmental, like sewage sludge, and biol., like mussel tissue and rice flour, after their rapid microwaveassisted digestion. The systematic study of the effect of six different matrix modifiers demonstrated that, using an ashing temp. of 1000° and carrying out the atomization at 2700°, neither modifier nor background correction are necessaries. The recommended procedure is based on the pressurized acid digestion of samples with aqua regia, HF and H2O2, in different steps, inside hermetically closed teflon reactors, heated by radiation in a microwave oven, at power levels between 330 and 550 W. the complete dissoln. of samples, Ni is detd. by ETAAS carrying out the absorbance measurements in the peak height mode, and using ag. solns. of Ni as stds.
- L12 ANSWER 54 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 126:211169 CA
- TI Microwave pressure digestion development, performance, and safety of a novel system of sample preparation
- AU Kettisch, P.; Schalk, A.; Knapp, G.; Zischka, M.
- CS Anton Paar KG, Graz, A-8054, Austria
- SO CANAS '95, Colloquium Analytische Atomspektroskopie, Konstanz, Germany, Apr. 2-7, 1995 (1996), Meeting Date 1995, 355-359. Editor(s): Welz, Bernhard. Publisher: Bodenseewerk Perkin-Elmer, Ueberlingen, Germany.
- LA German
- AB An overview is given of the Multiwave system for microwave pressure digestion (≤80 bars for quartz reaction vessels, ≤40 bars for PFA vessels; ≤300°). The efficiency of the system is enhanced by sep. control of pressure for different reaction vessels. Reaction temp. is monitored by using an IR system. The system is appropriate for the detn. of heavy metals in food samples.
- L12 ANSWER 56 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 126:152057 CA
- TI Direct processing and analysis of solid and other complex samples with automatic flow injection systems
- AU Zhi, Zheng-liang; Rios, Angel; Valcarcel, Miguel
- CS Dep. Analytical Chem., Univ. Cordoba, Cordoba, 14004, Spain
- SO Critical Reviews in Analytical Chemistry (1996), 26(4), 239-260
- AB A review, with 90 refs., is given. The use of a suitable sampling or sample-processing unit connected to a flow injection system can significantly expand the scope of application of the flow injection technique by enabling the direct introduction and treatment of solid and other complex samples in a single automated system. In recent years various sample pretreatment techniques, including electrolytic dissoln., online leaching, continuous microwave digestion, online sterilizable membrane dialysis/filtration, pervaporation, dynamic gas-extn., and flow-reversal liq.-liq. extn., among others, proved useful for solving a variety of anal. problems in conjunction with flow injection systems. The principles behind these techniques and their applications in fast assay and online process monitoring are reviewed and discussed. Selected major advances in this research field are outlined.

- L12 ANSWER 57 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 126:98532 CA
- TI Determination of Environmental Lead Using Continuous-Flow Microwave

  Digestion Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
- AU Beary, Ellyn S.; Paulsen, Paul J.; Jassie, Lois B.; Fassett, J. D.
- CS Chemical Science and Technology Laboratory, National Institute of Standards and Technology, Gaithersburg, MD, 20899, USA
- SO Analytical Chemistry (1997), 69(4), 758-766
- AB A com. flow-through microwave system was successfully applied as an alternative sample prepn. procedure for lead detns. using isotope diln. inductively coupled plasma quadrupole mass spectrometry. Sample is introduced as a slurry and then injected into a carrier stream which is continually flowing through the system. As configured, the sample dissoln. is sequential, taking ~10 min/sample. This closed system is easy to use and produces low blanks, thus providing a viable alternative to the direct introduction of untreated samples, which can degrade anal. results. Leaves, air filters, urine, sludge, dust, and paint std. ref. materials previously certified for lead using isotope diln. mass spectrometry (IDMS) were used to evaluate the accuracy of this automated sample prepn. device. Lead concns. in the nanograms to micrograms per g range were accurately detd. when compared with the certified value and previous IDMS results.
- L12 ANSWER 58 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 126:79426 CA
- TI Wastewater treatment system
- PA Wastech International, Incorporated, USA; Marchesseault, Guy; Rummler, John; Sauk-Schubert, Heinz; Beal, Thomas; Mchenry, Steven
- SO PCT Int. Appl., 110 pp.
- PΤ WO 9637440 A1 19961128 WO 1996-US6964 19960522 US 5725762 Α 19980310 US 1995-445726 19950522 US 5843304 Α 19981201 US 1995-445686 19950522
- PRAI US 1995-445686 A 19950522
- AB A treatment system is provided which can treat solid materials and/or liq. materials. Preferably, a separator initially separates liq. and solid materials, and the **solid** materials are deposited in a **microwave reaction** chamber to be subjected to **microwaves**. The liqs. exiting the separator are preferably also passed through a **filter** assembly, which retains any suspended particles remaining in the liq., and subjects the particles to microwaves. Exhaust **filter** can also be provided for the **microwave reaction** chamber.
- L12 ANSWER 62 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 125:157190 CA
- TI Use of a microwave oven for digestion of analytical samples before determination of metals by atomic absorption spectrometry (AAS)
- AU Przygoda, Dorota; Dittwald, Ewa; Samsonowska, Katarzyna; Kalinowski, Krzysztof
- CS Instytut Chemii Przemyslowej, Warsaw, Pol.
- SO Chemik (1996), 49(5), 125-126
- LA Polish
- Procedures for microwave digestion of selected vegetable materials, cosmetics, salts and esters of higher fatty acids, galvanic wastes and catalysts are elaborated. For the digestion and mineralization of samples a CEM company microwave oven type MDS-2000 equipped with translucent PFA containers with TPFE inserts was used. In the applications mentioned above a considerable time redn. were obtained, esp. in the detn. of metals in

mixts. with low- and high-boiling org. compds. contg. Cr salts of org. acids.

- L12 ANSWER 67 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 124:337083 CA
- TI Microwave digestion using dual PTFE containers for analysis of trace elements in small amounts of biological samples
- AU Mizushima, Ryuji; Yonezawa, Mitsuru; Ejima, Akiko; Koyama, Hiroshi; Satoh, Hiroshi
- CS Nippon General Trading Co., Tokyo, 103, Japan
- SO Tohoku Journal of Experimental Medicine (1996), 178(1), 75-9
- AΒ The anal. of trace elements in biol. samples is essential to extend our knowledge on human health and disease. Inductively coupled plasma mass spectrometry (ICP-MS) makes it possible to simultaneously det. these elements in trace amts. Before anal., however, biol. samples such as organs and tissues must be liquefied and extra org. materials must be decompd. by acid digestion. We established a method of microwave digestion using dual PTFE containers to minimize the amt. of samples. Samples (35-45 mg) of std. ref. materials, bovine liver (1577a, NIST) and fish flesh (MA-A-2, IAEA), were weighed in PTFE-PFA vials and a small amt. of nitric acid (0.5 mL) was added. The vials were sealed and two PTFE-PFA vials were placed in a PTFE-TFM vessel contg. 6 mL of pure water. Then the vessels were placed in a rotor and the samples were digested for 38 min in a microwave oven according to a pre-set program. After the program was completed, the samples were analyzed by ICP-MS. The detd. values of elements of the microwave-digested samples matched the certified values of the std. ref. materials. Therefore, the digestion using dual containers was successfully applied to small samples.
- L12 ANSWER 68 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 124:330955 CA
- TI Application of inductively coupled plasma mass spectrometry for total metal determination in silicon-containing solid samples using the microwave-assisted nitric acid-hydrofluoric acid-hydrogen peroxide-boric acid digestion system
- AU Wu, Shaole; Zhao, Yu-Hui; Feng, Xinbang; Wittmeier, Adolph
- CS Environ. Chem., Alberta Environ. Cent., AB, T9C 1T4, Can.
- SO Journal of Analytical Atomic Spectrometry (1996), 11(4), 287-96
- AB The microwave-assisted conventional HNO3-HF-H2O2-H3BO3 digestion system was explored for direct ICP-MS anal. of total metals in silicon-contq. solid samples. In closed Teflon PFA vessels under microwave heating with temp./pressure regulation, a 0.25 g portion of sample contg. ≤33% silicon was digested in 1.5 mL of HF (48%), 5 mL of concd. HNO3 and 2 mL of H2O2 (30%), followed by a 2nd digestion stage with 12 mL of 5% m/v boric acid. The amt. of HF used was approx. twice the stoichiometric requirement for 33% silicon contained in the samples, assuming all silicon exists as silica. The amt. of boric acid used was the stoichiometric requirement for the HF With a sample diln. factor of 2000 (v/m), the end soln. contained ≤0.17% total dissolved solids for ICP-MS anal. The ICP-MS system was calibrated by the method of external stds. prepd. in reagent blank solns. with In as the internal std. The sensitivity of the In signal in a 5-8 h operation was only decreased by 5-10%. Background interferences from the developed is simple and rugged and suitable for routine anal. of at least 25 elements: Al, Ag, As, Ba, Bi, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Ni, Pb, Sb, Sn, Sr, Th, Ti, Tl, U, V and Zn. The recoveries for most of these elements in std. ref. materials including coal fly ash, urban particulate matter, sediment and soil were within 90-110%, and the relative sediment and

soil were within 90-110%, and the relative std. deviations were within 5%.

The method cannot det. B and is not suitable coal fly ash, urban particulate matter, sediment and soil were within 90-110%, and the relative std. deviations were within 5%. The method cannot det. B and is not suitable for Hg detn. The detection limits for Be and Se detn. are not adequate.

- L12 ANSWER 69 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 124:330921 CA
- TI The comparison of **sample preparation** techniques for the determination of technetium-99 in pure uranium compounds and subsequent analysis by inductively coupled plasma-mass spectrometry (ICP-MS)
- AU Makinson, Peter R.
- CS Chemical and Metallurgical Services Department, British Nuclear Fuels plc., Preston/Lancashire, PR4 OXJ, UK
- SO ASTM Special Technical Publication (1995), STP 1291(Applications of Inductively Coupled Plasma-Mass Spectrometry to Radionuclide Determinations), 7-19
- In this presentation, a procedure for the detn. of ng/g levels of AΒ technetium-99 in uranium dioxide powder, hydrolyzed uranium hexafluoride (Hex), and uranyl nitrate liquor (UNL) is discussed, and three methods for the dissoln. of uranium dioxide are compared. Digestion of uranium dioxide using concd. nitric acid in a stoppered glass tube gives variable results. A concd. hydrochloric acid dissoln. using microwave digestion in sealed TFEfluorocarbon PFA digestion vessels gives reproducible results, as does the digestion using dil. nitric acid in open beakers. The effects of the isobaric interference of ruthenium-99 and the mol. interference of 98MoH and the use of suitable algorithms to correct for such interferences are Compensation of signal suppression due to the uranium matrix is made using matrix-matched calibration std. solns. and instrumental drift is monitored and cor. by the addn. of rhodium as an internal std. A detection limit (2 s) of 0.3 ng g-1 U is achieved using a soln. contg. 2 g of uranium per L and a 30-s per isotope acquisition time, using peak jump mode.
- L12 ANSWER 73 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 124:210840 CA
- TI Trace metal analysis of coal fly ash collected plain and on a quartz fiber filter
- AU Laitinen, T.; Revitzer, H.; Tolvanen, M.
- CS VTT, Chem. Technol., Environ. Technol., Espoo, FIN-02044, Finland
- SO Fresenius' Journal of Analytical Chemistry (1996), 354(4), 436-41
- As imple, uniform procedure has been developed for microwave-based digestion of fly ash samples collected from the hoppers of an electrostatic precipitator (ESP), or collected from flue gases on a heat-resistant quartz fiber filter and for subsequent at. absorption spectrometric anal. of trace metals (Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, V and Zn). HNO3, HCl, HF and mixts. have been tested as digestion acids. The combination of HNO3 and HF has been found to be efficient for the digestion of fly ash samples with and without quartz fiber filter material. In spite of the complicated matrix, results with satisfactory accuracy and precision (relative std. deviation below 10% for most of the elements) have been obtained. In the case of anal. of fly ash together with a quartz fiber filter, the calibration curve has to be detd. by std. addn. to a blank soln. contg. a dissolved blank quartz fiber filter.
- L12 ANSWER 74 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 124:183999 CA
- TI Multi-element analysis of airborne particulate matter by various spectrometric methods after microwave digestion
- AU Wang, Chu-Fang; Yang, Jenq-Yann; Ke, Chenq-Hsiung

- CS Institute of Nuclear Science, National Tsing Hua University, Hsinchu, 30043, Taiwan
- SO Analytica Chimica Acta (1996), 320(2-3), 207-16
- An anal. method using microwave digestion procedure in combination with various spectrometric methods, including at. absorption spectrometry, inductively coupled plasma at. emission spectrometry, and inductively coupled plasma mass spectrometry, was developed for the multi-element detn. in airborne particulate matter collected on PTFE filters by a dichotomous sampler. In order to achieve more sensitive and rapid multi-element analyses, special PTFE-lined digestion vessels were used. It was found that complete digestion of airborne particulates with an acid mixt. of HNO3-HClO4-HF (3:7:1, vol./vol./v) can be achieved in the microwave-irradiated closed vessel system and direct spectroscopic measurement of the digested sample after appropriate diln. A recovery study was conducted using a multi-element std. and NIST Std. Ref. Material 1648 Urban Particulate. Sixteen major, minor, and trace elements in airborne particulate matter were detd.
- L12 ANSWER 76 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 123:295015 CA
- TI Analytical procedures on multi-element determinations of airborne particles for receptor model use
- AU Wang, C. F.; Chang, E. E.; Chiang, P. C.; Aras, N. K.
- CS Institute Nuclear Science, National Tsing Hua University, Hsinchu, Taiwan
- SO Analyst (Cambridge, United Kingdom) (1995), 120(10), 2521-7
- AB A multistage anal. procedure is developed in this investigation to det. 22 elements (Al, As, Ba, Ca, Cd, Cl, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, S, Sb, Si, Sr, Ti, V, and Zn) on loaded PTFE-filter samples of atm.-particulate matter collected by dichotomous samplers. It includes a direct x-ray fluorescence measurement followed by instrumental neutron activation anal. (INAA) or microwave acid digestion of the sample for spectroscopic anal. ICP-MS was employed for elemental anal. after the digestion. A combination of different anal. methods and std. operational procedures were developed to meet the requirements for receptor model anal. The quality assurance program includes the intercomparison with the use of alternative methods for accuracy and precision control.
- L12 ANSWER 79 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 123:217229 CA
- TI Microwave digestion and alkali fusion procedures for the determination of the platinum-group elements and gold in geological materials by ICP-MS
- AU Totland, Marina M.; Jarvis, Ian; Jarvis, Kym E.
- CS School of Geological Sciences, Kingston University, Penrhyn Road, Kingstonupon-Thames, Surrey, KT1 2EE, UK
- SO Chemical Geology (1995), 124(1-2), 21-36
- Inductively coupled plasma-mass spectrometry (ICP-MS) is an ideal technique for detg. the Pt-group elements (PGE's) and Au, with rapid data acquisition and low detection limits of 0.03-0.22 ng mL-1. Two methods are described for the prepn. of geol. samples using microwave digestion prior to detn. of the PGE's and Au by ICP-MS. In one method, 0.5-g samples are dissolved in sealed all-PFA microwave digestion vessels using HNO3-HCl-HF-HClO4 acids. Samples are transferred to open PTFE beakers and evapd. to incipient dryness, final solns. being taken up in 1 M HCl prior to anal. The method was evaluated using a suite of well-characterized international ref. materials (RM's). In some cases, the method resulted in a complete digestion and quant. data were obtained for Rh, Pd, Ir, Pt and Au, although insufficiently low limits of detn. precluded the detn. of all five elements in all materials. In other cases, an insol. residue remained and recoveries

of the PGE's and Au were dependent on the element concerned and the mineralogy of each sample. A 2nd method employs 1-g samples and microwave digestion with aqua regia-HF, in higher-pressure Ultem-jacketed Teflon PFA sealed-vessels. Samples are subsequently evapd. to near dryness, digested in 0.5M HCl, filtered, and the insol. residues are fused with small quantities of 1:1 Na2O2+Na2CO3 or Na2O2, before being dissolved in 0.5M HCl. The combined solns. are analyzed by ICP-MS. Data obtained for a wide range of RM's showed good agreement with ref. values. Both methods provide viable means of quantifying Ru, Rh, Pd, Ir, Pt and Au in mineralized samples, but both are limited by modest lower limits of detn. in samples of 0.2-1  $\mu \rm g$  g-1. Only the combined microwave digestion-minifusion technique yields fully quant. data for samples contg. refractory minerals.

- L12 ANSWER 80 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 123:142049 CA
- TI Manganese and Zinc Analysis in Milk by **Microwave** Oven **Digestion** and Platform Graphite Furnace Atomic Absorption Spectrometry
- AU de la Fuente, Miguel Angel; Guerrero, Gonzalo; Juarez, Manuela
- CS Instituto del Frio (CSIC), Ciudad Universitaria s/n, Madrid, 28040, Spain
- SO Journal of Agricultural and Food Chemistry (1995), 43(9), 2406-10
- AB A conventional microwave oven acid digestion system employing PTFE reactors was tested for sample digestion prior to the anal. of Mn and Zn in milk. The digestion procedure was based on sample dissoln. with nitric acid using microwave heating in closed vessels. Sample treatment time in the microwave oven was reduced to 15 min. The resulting solns., analyzed for Zn by flame at. absorption spectrometry (FAAS) and by platform furnace at. absorption spectrometry with Zeeman background correction for Zn and Mn, showed good agreement with the certified milk values (SRM-1549 nonfat milk powder, National Institute of Stds. and Technol.), and results were comparable to those obtained by dry mineralization in real samples. The precision of the method was about 6% for Mn and 5% for Zn by graphite furnace at. absorption spectrometry (GFAAS) with limits of detection of 0.07 and 0.28  $\mu$ g/L, resp. The digestion procedure can be used to det. In by FAAS and Mn by GFAAS in milk anal. Using GFAAS, Zn can also be measured in milk fractions where it is found in smaller concns.
- L12 ANSWER 83 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 123:39703 CA
- TI Microwave decomposition for airborne particulate matter for the determination of trace elements by inductively coupled plasma mass spectrometry
- AU Wang, C. F.; Chen, W. H.; Yang, M. H.; Chiang, P. C.
- CS Inst. Nucl. Sci., Natl. Tsing Hua Univ., Hsinchu, 30043, Taiwan
- SO Analyst (Cambridge, United Kingdom) (1995), 120(6), 1681-6
- AB A microwave digestion method utilizing an acid mixt. of HNO3-HClO4-HF (3 + 5 + 2 vol./vol.) in closed vessels was developed for the detn. of major, minor and trace elements in very small amts. of airborne particulate matter collected on Teflon filters by a dichotomous sampler, and was tested on NIST Std. Ref. Material (SRM 1648 Urban Particulate Matter). Analyses were carried out by inductively coupled plasma mass spectrometry (ICP-MS). Sixteen elements (Al, As, Ba, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V and Zn) were detd. The results generally showed high reproducibility and good agreement with the certified values, except for As and Cr. The feasibility of applying the microwave digestion pre-treatment with ICP-MS to real samples was also examd. It was demonstrated that the suggested method is more suitable than conventional methods for real sample anal.

- AN 121:270645 CA
- TI Application of pressurized **sample preparation** methods for analysis of steels and copper alloys
- AU Borszeki, Janos; Halmos, Pal; Gegus, Erno; Karpati, Peter
- CS Dep. Anal. Chem., Univ. Veszprem, Veszprem, H-8201, Hung.
- SO Talanta (1994), 41(7), 1089-93
- AB Pressurized sample prepn. devices (High Pressure Asher, Pressurized Microwave Digestion system, compared with a PTFE decompn. vessel) were used to dissolve certified metal alloy samples (steel, Cu) for ICP anal. Based on the results of the anal. both up-to-date devices can be advantageously applied to quickly and quant. dissolve metal alloy samples. To dissolve the samples, two different kinds of acid mixts. (A: nitric and HCl; B: nitric and hydrochloric and sulfuric and H3PO4) were used. The sample prepn. is simpler and less time-consuming than the earlier commonly used methods, sample loss and degree of contamination are also reduced. Steel samples contg. W, Ti and Nb (<0.5%) can only be analyzed using a mixt. of the four acids. By dissolving steel samples in the nitric and HCl mixt., the concn. of their most common elements (Cr, Ni, Mn, V, Cu) as well as their S and P content can be detd. Cu alloy samples can be dissolved quickly by the pressurized microwave decompn. device using HCl and dild. (1:1) HNO3.
- L12 ANSWER 93 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 120:123820 CA
- TI Development of a high pressure/temperature focused microwave heated **teflon** bomb for **sample preparation**
- AU Matusiewicz, Henryk
- CS Dep. Anal. Chem., Politech. Poznanska, Poznan, 60-965, Pol.
- SO Analytical Chemistry (1994), 66(5), 751-5
- AB A new high pressure/temp. TFM-Teflon bomb for anal. sample prepn. is described. The novel prototype system uses focused microwaves, operated at 2.45 GHz, to improve digestion capability. Up to 100-W microwave power can be concd. into a single polymer deriv. vessel contg. sample and HNO3. Methodol. was developed using powd. biol. ref. material. The feasibility of using H2O for in situ vessel cooling was studied. The residual C content of bovine liver sample was detd. by coulometry after combustion in an O stream to evaluate the effectiveness of the decompn. procedure. With this new decompn. device, org. material is totally oxidized with HNO3 in a single-step procedure. The sample prepn. time is ~10 min (including subsequent cooling time and prepn. of the final soln.).
- L12 ANSWER 95 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 119:208879 CA
- TI High-pressure microwave dissolution of ceramics prior to trace metal determinations by microwave induced plasma atomic emission spectrometry
- AU Matusiewicz, Henryk
- CS Dep. Anal. Chem., Politech. Poznanska, Poznan, 60-965, Pol.
- SO Mikrochimica Acta (1993), 111(1-3), 71-82
- AB A com. lab. microwave acid digestion system was evaluated for the acid dissoln. of ceramic powders (Al2O3, AlN, BN and Si3N4) prior to the detn. of their trace element content by microwave induced plasma at. emission spectrometry. Newly designed vessels, capable of withstanding internal pressures of over 110 bar, provide rapid and satisfactory results for sample dissoln. Sample prepn. time was approx. 30 min (including the subsequent cooling time and prepn. of the final soln.). Results from conventional stainless-steel acid digestion vessel (Teflon bomb) dissoln. are compared with the microwave bomb results of microwave plasma at. emission spectrometry.

- L12 ANSWER 100 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 118:3368 CA
- TI Factorial design approach to microwave dissolution
- AU Mohd, A. A.; Dean, J. R.; Tomlinson, W. R.
- CS Sch. Appl. Sci., Inst. Teknol. Mara, Shah Alam, 40000, Malay.
- SO Analyst (Cambridge, United Kingdom) (1992), 117(11), 1743-8
- AB A fractional factorial design was used to explore the variables that affect microwave dissoln. using perfluoroalkoxy (PFA)-Teflon digestion vessels. Optimum operating conditions for Nat. Research Council of Canada, certified ref. material TORT-1 Lobster Hepatopancreas and Nat. Inst. of Stds. and Technol., Std. Ref. Material (SRM) 1575 Pine Needles, were obtained by this procedure. The optimum conditions found for each variable are: 0.25 g sample mass, 6 mL concd. HCl, 6 mL concd. HNO3, 3 mL concd. HF, and 90% microwave power for a total dissoln. time of 15 min. Results of Ca, Fe, Cu, and Zn in TORT-1 and Ca and Fe in SRM 1575 were in agreement with the certified values.
- L12 ANSWER 104 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 117:107632 CA
- TI Microwave digestion of biological samples with acid mixture in a closed double PTFE vessel for metal determination by "one-drop" flame atomic absorption spectrometry
- AU Kojima, I.; Kato, A.; Iida, C.
- CS Lab. Anal. Chem., Nagoya Inst. Technol., Nagoya, 466, Japan
- SO Analytica Chimica Acta (1992), 264(1), 101-6
- AB A microwave-heat system using a closed double PTFE vessel with a polypropylene jacket was evaluated for rapid sample prepn. for the detn. of trace elements in biol. samples. The microwave-assisted digestion was based on sample dissoln. with a HNO3-HClO4-HCl-HF mixt. The simultaneous digestion of six samples including one blank was completed within 15 min even with very mild heating. The presence of an alk. soln. between the PTFE vessels was very effective in aiding the digestion and absorption of evolved acid gases. Six metals (Ca, Cu, Fe, Mg, Mn and Zn) in std. ref. materials, NIST-SRM 1577 Bovine Liver, NIES-CRM No. 1 Pepperbush, No. 6 Mussel, No. 5 Human Hair, No. 7 Tea Leaves and No. 9 Sargasso, were analyzed by one-drop flame at. absorption spectrometry. Good agreement of the anal. results with the certified values was obtained.
- L12 ANSWER 110 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 115:202693 CA
- TI Rapid stopped-flow microwave digestion system
- AU Karanassios, Vassili; Liu, F. H.; Liu, B.; Salin, Eric D.
- CS Dep. Chem., McGill Univ., Montreal, QC, H3A 2K6, Can.
- SO Journal of Analytical Atomic Spectrometry (1991), 6(6), 457-63
- AB A prototype system for stopped-flow microwave-assisted wet digestions has been developed. A coiled Teflon perfluoroalkoxy tube serves both as a sample container and as a digestion vessel. A sample plug, consisting of a water slurry mixed with an acid mixt., is pumped into the coil. Sample flow is stopped, the coiled tube is sealed (by closing an input and an output valve), and microwave power is applied for 2 min for digestion of the sample. Methodol. was developed using powd. botanical ref. samples and was tested with powd. botanical and biol. ref. materials. The digests were analyzed by inductively coupled plasma at. emission spectrometry. In addn. to comparisons with certified values, the results were compared with those obtained by conventional open-vessel hot-plate digestions, by open-vessel microwave digestions, and by digestions taking 32 min by using the coiled tube system. Precise, and in many instances, quant. digestions were obtained using a net digestion time of 2 min. Elemental recoveries were

sample type and digestion time dependent and were comparable with, and sometimes, superior to those obtained when using a 3-h long hot-plate digestion. In this preliminary study, characteristics, limitations, and future directions are discussed.

- L12 ANSWER 115 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 115:63514 CA
- TI Vapor-phase acid digestion of inorganic and organic matrixes for trace element analysis using a microwave heated bomb
- AU Matusiewicz, Henryk
- CS Dep. Anal. Chem., Politech. Poznanska, Ponznani, 60-965, Pol.
- SO Journal of Analytical Atomic Spectrometry (1991), 6(4), 283-7
- AB A vapor-phase microwave pressure digestion technique employing a special polytetrafluoroethylene-based microsampling device was evaluated for the acid digestion of marine sediment and biol. tissue samples prior to the detn. of their trace and minor element content. Inorg. and org. constituents are almost completely solubilized by vapor-phase attack (with an HNO3-HF mixt. for the marine sediment and HNO3 for the marine biol. tissue) in a perfluoroalkoxy-Teflon pressure bomb. The residue was taken up in 0.5 mol dm-3 HNO3 and analyzed by flame and electrothermal at. absorption spectrometry. Good agreement between the results and certified values for 15 elements was found. The sample prepn. time was approx. 45 min for the biol. tissue and 90 min for the sediment (including the subsequent cooling time and prepn. of the final soln.).
- L12 ANSWER 118 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 114:239386 CA
- TI Application of the **microwave** acid **digestion** method to the decomposition of rock **samples**
- AU Suzuki, Toshitaka; Sensui, Mayumi
- CS Fac. Sci., Yamagata Univ., Yamagata, 990, Japan
- SO Analytica Chimica Acta (1991), 245(1), 43-8
- The microwave acid digestion method was applied to the decompn. of rock samples and optimum conditions were investigated. Samples of 10-100 mg were decompd. by changing the amt. and compn. of acid, heating time, and no. of reheating steps, and then the concns. of Si, Fe, Mn, Na, K and Mg in these samples were measured. The concns. agreed with reported values when 10 mg of sample were decompd. by heating for 60 s with 0.3 mL of concd. HNO3 and 0.1 mL of concd. HF. Similarly, 100 mg of sample were also decompd. successfully by heating for 45-110 s with 0.3-1.0 mL of concd. HNO3 and 0.4-0.7 mL of concd. HF. It is concluded that the microwave acid digestion method decomps. rock samples with a very short heating time and with small amt. of reagents compared with methods using conventional sealed PTFE vessels, which require several hours for the heating step and several milliliters of reagents.
- L12 ANSWER 125 OF 148 CA COPYRIGHT 2003 ACS on STN
- AN 113:223519 CA
- TI Practical options for microwave digestions
- AU Kimber, Graham M.; Kokot, Serge
- CS Queensland Univ. Technol., Brisbane, Australia
- SO TrAC, Trends in Analytical Chemistry (1990), 9(6), 203-7
- AB A review with 28 refs. Some options for the use of microwave ovens to facilitate acid digestion of various anal. samples are discussed. The use of inexpensive domestic ovens in conjunction with com. available polytetrafluoroethylene digestion vessels is highlighted. Various lab. practices are discussed and examples of successful acid digestion recipes are reported.

L12 ANSWER 129 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 113:108340 CA

TI Microwave acid digestion of geological and biological standard reference materials for trace element determination by inductively coupled plasma-mass spectrometry

AU Noeltner, Thomas; Maisenbacher, Peter; Puchelt, Harald

CS Inst. Petrogr. Geochem., Univ. Karlsruhe, Karlsruhe, D-7500/1, Germany

SO Spectroscopy (Duluth, MN, United States) (1990), 5(4), 49-53

- AB Rapid acid digestions of a variety of geol. and biol. samples are possible using microwave digestion techniques. To meet the anal. requirements and the extremely low blank background values necessary for accurate trace and ultratrace element detn. with inductively coupled plasma-mass spectrometry (ICP-MS), special polytetrafluoroethylene-based digestion vessels were used. The particular features of the microwave digestion system, including the automatic capping module and the gas exhaust module, were set up to minimize the possibility of error due to sample contamination. Anal. procedures and results of the expts. are reported.
- L12 ANSWER 138 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 111:93236 CA

TI Trace element analysis of biological material following pressure **digestion** with nitric acid-hydrogen peroxide and **microwave** heating

AU Matusiewicz, Henryk; Sturgeon, Ralph E.; Berman, Shier S.

CS Div. Chem., Natl. Res. Counc. Canada, Ottawa, ON, K1A OR9, Can.

SO Journal of Analytical Atomic Spectrometry (1989), 4(4), 323-7

- AB A com. microwave acid-digestion system employing three types of closed vessels, a pressure-relief PFA-Teflon bomb (A); a pressure-relief Berghof all PTFE bomb (B); and a completely closed Parr microwave acid digestion bomb (C), was evaluated for sample digestion prior to the detn. of trace elements in a biol. tissue. The digestion procedure was based on sample dissoln. with an HNO3-H2O2 mixt. using microwave heating. The resulting solns., analyzed by flame and graphite furnace at. absorption spectrometry, showed good agreement for all three closed-vessel techniques. Sample prepn. time was approx. 10 min for vessels B and C, and 15 min for vessel A (including subsequent cooling time and prepn. of the final soln.).
- L12 ANSWER 148 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 105:130099 CA

TI Microwave energy for acid decomposition at elevated temperatures and pressures using biological and botanical samples

AU Kingston, H. M.; Jassie, L. B.

CS Cent. Anal. Chem., Natl. Bur. Stand., Gaithersburg, MD, 20899, USA

SO Analytical Chemistry (1986), 58(12), 2534-41

AB A closed **vessel microwave digestion** system is described. In situ measurement of elevated temps. and pressures in closed Telfon **PFA** vessels during acid decompn. of org. samples is demonstrated. Temp. profiles for the acid decompn. of biol. and botanical std. ref. materials are modeled by the dissolving acid. Microwave power absorption of nitric, hydrofluoric, sulfuric, and hydrochloric acids is compared. An equation is applied to acid microwave interactions to predict the time needed to reach target temps. during sample dissoln. Reaction control techniques and safety precautions are recommended.